CONSOLIDATION OF HISTORICAL WOODS USING POLYVINYL BUTYRAL/ZINC OXIDE NANO-COMPOSITE: INVESTIGATION OF WATER ABSORPTION, WETTABILITY, AND RESISTANCE TO WEATHERING

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Abstract

Polyvinyl butyral is a consolidate that mostly used for consolidation of degraded and weak woods. The aim of present research is to study the efficiency of polyvinyl butyral/zinc oxide nanocomposites as consolidant of old dried woods and evaluate the influence of this material on penetration and wettability of wood and also durability against accelerated aging of Plantenus Orientalis wood samples. All wood samples used in this research are belonged to Qajar period and were obtained from the same piece in equal and relatively well condition and held in a chamber of controlled temperature and relative-humidity to maintain their weight after they were cut into certain cross-sections. In this research, 0.5, 1.0 and 1.5 wt% of zinc oxide was added to the matrix of 10% polyvinyl butyral using the whole immersion method. The efficiency of consolidating was examined by simple weighting method, and wettability of samples was evaluated by contact angle test. The values of the degradation rate of samples after accelerated aging were measured by FTIR-ATR (Fourier-Transform Infrared Spectroscopy-Attenuated Total Reflection) methods. The results showed that introduction of 0.5 and 1 wt% of nano-zinc oxide to the consolidate polymer matrix led to a decrease in water penetration and wettability of samples. On the other hand, the degradation rate in the accelerated aging condition of samples which were treated with both polyvinyl butyral and 1.5 wt% dispersion of nano- zinc oxide was lower in comparison with other samples.

Keywords: Wood; Consolidate; Nano-Composite; Polyvinyl Butyral; Zinc Oxide; Water Absorption

Introduction

Wood is undoubtedly one of the most important materials among several fundamental architectural elements. Wooden parts have been extensively used as structural components in various parts of buildings as in movable works. Arising from their special characteristics such as heterogeneity and changeable dimensions in specific conditions, wooden materials have low resistivity against environmental agents and are thus susceptible to degradation [1].

Therefore, wooden artifacts having historical or archeological importance should be protected from environmental threats and potential degradation. Consolidation is a conventional procedure for the conservation of degraded wooden artifacts [2]. Generally, the purpose of consolidation is to restore cohesion, physical properties and mechanical strength of wooden artifacts as far as their authenticity is not treated [3-4].

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In every consolidation procedure, there are some key factors that affect the efficiency of the procedure such as consolidate material, solvent type, solution concentration and the method of application. Adjustment of these factors highly depends on the type and state of the artifact's components and the nature of case study in research areas [5]. A wide variety of materials are being used for consolidation; however, solutions are the most important type of consolidates [4, 6-7]. Among various thermoplastic resins which are commonly used as consolidates, Paraloid B72 and Poly Vinyl Butyral (PVB) are known as the most effective ones for consolidation of wooden artifacts [7-10]. However, PVB has attracted the most attention for this purpose owing to its desirable and effective features [8]. So, promotion of this solvent for the consolidation purposes can be a considerable improvement in conservation measurement. A common approach in promoting the strength of polymers is adding inorganic nanoparticles and many efforts have been done in this field [11-15].

Also, several efforts have been undertaken for consolidation of wooden artifacts by nanoparticle-filled polymers [5, 16-18]. On the other hand, many efforts have been made to improve the biological properties of polyvinyl butyral in order to be applied in wood treatment processes. [19] However, no research study has focused on the behavior of PVB in the presence of water or in a humidified atmosphere.

Nevertheless, many studies have investigated the influence of water and humidity on the physical properties of wooden parts [20-22]. The attack of micro-organisms mainly depends on the moisture of cell wall of the wood, and water absorption depends on density and water emission factor in the wood [23]. The use of synthetic polymers in the consolidation of wood can influence the rate of water absorption as well as elongation and swelling rate [24]. Waterproofing of wooden goods can be achieved through a coating of wood by inorganic compounds [25-27], synthetic resins [28] and other hydrophobic agents. Nowadays ZnO is broadly used in nanotechnology science fields to enhance the operation of materials and devices through different techniques in nanometer scale [29]. In the last decade, the nanostructured ZnO has attracted the attention of numerous researchers due to its outstanding physical, chemical and sensing properties [30, 31]. Also an increasing number of scientific reports have appeared on using ZnO in photocatalytic degradation of organic compounds [32]. ZnO has been, in general, recognized as safe (GRAS) material category by the US Food and Drug Administration [31]. Regarding the above-mentioned aim, this research seeks to study the water stability and weathering resistance of poly vinyl butyral and zinc oxide nanocomposite in Plantenus wood samples and to evaluate this material as a consolidate for dry historical wooden artifacts through performing some related tests and analyzing the obtained results.

Experimental

Wooden Samples

In this research samples were obtained from the wooden stakes of a door of a building in Birjand in the east of Iran belonged to Qajar period. Samples were found in a same, relatively sound condition. Radial, tangential, and transversal sections were cut respectively in 10, 20 and 30mm [33]. After sanding with sandpaper H 240, samples were placed in a chamber in the laboratory (200±2°C, 50±5% RH) for 30 days to achieve weight stability and being prepared for treatment [6]. Platanus Orientalis-L type was chosen because it is local to Iran [34, 35]. It has been employed in many wooden artifacts, particularly in historical buildings [36-39] and plays an important role in Iranian wood art.

Consolidate Solutions

In this research polyvinyl butyral (as the polymer matrix) and zinc oxide nano-particles (as the filler) have been combined and used for consolidation of the wooden artifacts. For making the nano composite, PVB (ACROS) and zinc oxide nanoparticles with the size of 29 nm and 99.983% purity (research" trademark made in Spain) were used. PVB was diluted using

ethanol and 10% PVB solution was prepared. Different concentrations of nano-zinc oxide (0.5, 1.0 and 1.5%) were added to PVB solutions separately and were dispersed using ultrasonic homogenizer. Numbers of consolidate solutions used in this research are shown in Table 1.

Table 1. Samples code.

Samples Code	Soluble Compound
A	Untreated Sample
A1	PVB 10% in Ethanol
A2	A1+ 0.5 % Nano ZnO
A3	A1+ 1 % Nano ZnO
A4	A1+ 1.5 % Nano ZnO

Treatment Process

Dried samples having constant weight were immersed in the consolidate solutions for 24 hours and weighted afterward [33]. According to the difference between density values of wood and the solution, samples were suspended in the solution using a pin and stainless wire to avoid the movement of samples and to make a uniform condition and constant contact of the solution on the wood surface. Samples were held in a plumbed (plasticized) box during the treatment.

Water Absorption Test

Water absorption test was performed following ASTM (D7334-08) standard [40]. First, treated samples were weighted after solvent evaporation, and then immersed completely in distilled water and weighted again after different certain time periods (1, 2, 3 and, 24 hours). The values of water absorption (WA) for each sample were measured by equation (1) as follows:

$$WA(\%) = 100 \times (m_i - m_f)/m_f, \tag{1}$$

were: m_i - weight of samples after immersion and m_f - weight of samples before immersion.

Wetting Properties: Contact Angle

Water contact angle (4μL) was measured by a system equipped with a CCD camera capable of taking photos from water droplets and software designed to calculate contact angle statically. The test was performed in three points in three cross-sections of the samples (longitudinal, tangential and radial) following ASTM (D7334 – 08/2013) standard [41] was built with the Dataphysics, OCA 15 Plus system. The calculation of the angle between the tangent line of the drop and the line parallel to the film surface reveals the contact angle [42]. The contact angle of wood with distilled water was examined on both sides of the water droplet, and then average values were calculated for every droplet. This test was done on every cross-section of the samples.

Evaluation of Accelerated Aging using FTIR technique

The accelerated aging process was tested in order to investigate the changes that occur during the process in wood samples. The samples for temperature and humidity aging test were kept in an oven at 600ºC and 75±5% RH for 30 days. Afterward, to evaluate the protective effect of the treating material against UV light and to study the structural changes, the samples were kept inside a controlled compartment under UV lamp with the power of 18W and wavelength of 356nm for 120h [19]. Any structural changes caused by accelerated aging before and after the process were evaluated by FTIR-ATR spectrometer Tensor 27 by Bruker.

Statistical Analysis

Analysis of the results obtained from water absorption and wettability tests was performed through an accidental statistical plan under the test of variance analysis using SPSS software. Finally, Duncan's Multiple Domain test at a confidence level of 99% of average comparing was used.

Result and Discussion

Water Absorption

The results obtained from water absorption test are presented in Figure 1. One-way variance analysis reveals that there are meaningful differences in water absorption values of samples before and after treatment. The highest water absorption value, 70.14%, was obtained for untreated sample named as A. After addition of 0.5wt% nano-zinc oxide to sample, named as A2, water absorption was reduced by 10% compared to sample A1 (the sample without any nano-zinc oxide) and reached to 25.47%. Addition of 1wt% nano-zinc oxide to the sample, named as A3, decreased water absorption to 31% that is a considerable decrease as compared with A1. However, water absorption of A3 was increased compared with A2. The obtained results show that addition of low amounts (0.5 or 1.0wt%) of nano-zinc oxide to polyvinyl butyral solution has the desirable impact of reducing water absorption of *Platanus* wood samples.
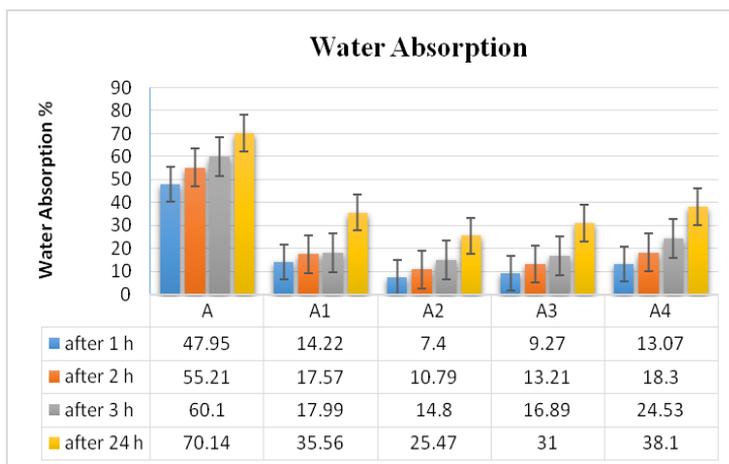

Water Absorption

	A	A1	A2	A3	A4
after 1 h	47.95	14.22	7.4	9.27	13.07
after 2 h	55.21	17.57	10.79	13.21	18.3
after 3 h	60.1	17.99	14.8	16.89	24.53
after 24 h	70.14	35.56	25.47	31	38.1

Fig. 1. Water Absorption (%) in the treated Plantenus wood samples at different time periods.

Wetting Capability

The results of wetting capability in three radial, tangential and longitudinal cross-sections are shown in figure 2. The samples treated with polyvinyl butyral/zinc oxide nanocomposites show better hydrophobicity compared with samples treated with polyvinyl butyral solution (A1), except for sample A4. It is worthy to note that all samples (A1, A2, A3, and A4) have had a lower contact angle in tangential section compared with the longitudinal and radial section, and thus, it may be concluded that *Platanus* wood is more hydrophilic in tangential section. It was observed that A2 sample showed an increase in contact angle in all cross-sections in comparison with A1 sample (nano-free) which demonstrate an increase in hydrophobicity. The results obtained from this test showed that hydrophobicity of wood samples will be promoted in all cross-sections by adding specific values of nano-zinc oxide to polyvinyl butyral solution. It should be noted that this property obviously depends on the amount of zinc oxide. In another word, by adding 0.5wt% nano-zinc oxide to the solution, the resistant of the wood sample to water increases, and by increasing this amount to 1%, this resistivity rises to the higher level (compared with nano-free solution) particularly in tangential and radial cross-section. However, as observed in sample A4, after addition of 1.5wt% of nano-zinc oxide to polyvinyl butyral solution, the hydrophobicity of wood sample was reduced (Table 2).
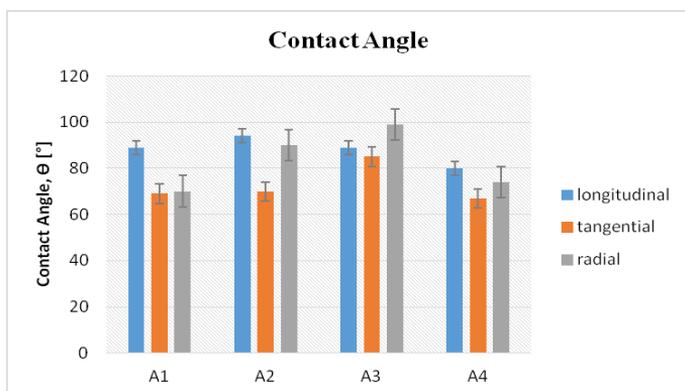
Fig. 2. The contact angles of treated samples in different cross-sections.

**Table 2**. The Contact angle for treated samples in different cross-sections.

Sample	Longitudinal	Tangential	Radial
A1	89±5	69±5	70±5
A2	94±5	70±5	90±5
A3	89±5	85±2	99±2
A4	80±5	67±2	74±1

### Evaluation of Accelerated Aging using FTIR

Generally, the presence of an absorption peak at 3300-3400cm^{-1} area is related to stretching vibration of O-H bonds of inner molecular bonds in cellulose [43, 44], the peak at 2923 cm^{-1} is related to vibration of aliphatic stretching C-H bonds of cellulose molecule and also to absorption of C-H bonds existing in methoxy of lignin molecules. On the other hand, the absorption peak at 2900cm^{-1} can be assigned to stretching vibrations of C-H bonds that are not changed by time, temperature or different conditions. The absorption peak at 1733cm^{-1} is an indicator of presence free C=O groups [45-47] in hemicellulose and confirms that oxidation occurred in the cellulose structure. Also, the absorption peaks at 1504 and 1593cm^{-1} can be attributed to the stretching vibrations of C=C double bonds belonging to lignin aromatic rings in the wood structure (this absorption is known for lignin). The absorption peaks at 1422 and 1234cm^{-1} are assigned to the flexural vibrations of OCH$_3$ methoxy groups of lignin molecules caused by deformation of C-H bonds in carbohydrates. The absorption peaks at 1330 and 1268 cm^{-1} are appeared due to the vibrations of specific unites of guaiacylpropane (G) in needle leaf trees and syringylpropane (S) in broadleaf trees. It should be noted that the absorption band at 1328cm^{-1} demonstrates a part of all structural components of wood since this band is related to C-H flexural motions of polysaccharides and G rings of lignin [46, 47]. The absorption peak at 1032cm^{-1} can be ascribed to stretching vibrations of C-O and C-C in cellulose and hemicellulose structure [19, 43-55]. On the other hand, the absorption peaks at 1370 and 1160cm^{-1} are characteristic of the carbohydrate structure (Figs. 3 and 4) [44].

Sample A1 showed water uptake of 35.56% after being immersed in distilled water for 24h. Such a low water uptake might probably be a consequence of pore filling ability of consolidates [5]. The rate of water absorption was further decreased to a greater extent after addition of nano-zinc oxide to the polymer solution. The same observation is also reported by [5], however, the authors noticed that higher loading amounts of nano-zinc oxide led to an increase in water absorption [5]. In a similar research study, the results obtained from wettability test were different in the three cross-sections of wood without any stable rate [5]. It can be explained that the wood properties are different in three tangential, radial and longitudinal sections, and thus, it would demonstrate different wetting capabilities [37]. Our results show that saturation of wood by polyvinyl butyral/zinc oxide nanocomposite containing

0.5 and 1.0wt% nanoparticles exerted a significant influence on water absorption of samples. The presence of nanoparticles on cell wall has first decreased wetting rate by reducing the access to free hydroxyl groups [13] and hence the reducing binding potential of wood with water. Second, the presence of nanoparticles on cell walls has noticeably decreased the size of cavities [57] and thus has disturbed permeation of water molecules through occupying some free space in pores of the wood and immobilization in open pores [58]. Therefore, water absorption and dimensional change of the wood would be remarkably reduced .
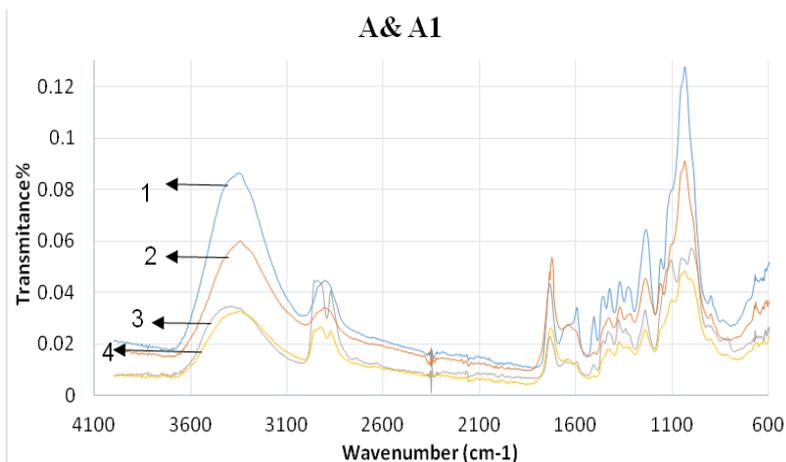
Fig. 3. FTIR spectra of samples A and A1:
1. A before aging, 2. A after aging, 3. A1 before aging), 4. A1 after aging
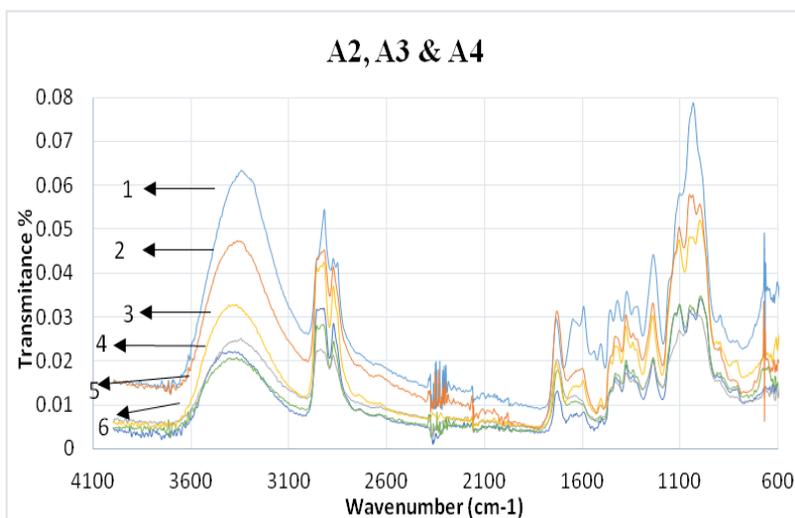
Fig. 4. FTIR spectra of samples A2, A3, and A4:
1. A3 before aging, 2. A3 after aging, 3. A2 before aging,
4. A2 after aging, 5. A4 after aging, 6. A4 before aging

An increase in the uptake (intensity of peaks) particularly in the range of 1650-1730cm[-1] confirms the oxidation and depolymerization of cellulose into secondary color groups (carbonyl) which is probably caused by decomposition of lignin molecules. Also, a decrease in the absorption intensity around 1504 and 1593cm[-1] in untreated samples after aging which is

characteristic to lignin absorption, related to C=C bonds, further proves the degradation of lignin [56]. Generally, absorption bands at 990-1100cm^{-1} and 2900cm^{-1} are characteristic of polyvinyl butyral. According to the obtained results, it can be claimed that addition of nano-zinc oxide to polyvinyl butyral somewhat improved resistant of the polymer to degradation. Moreover, it can be declared that the protection process becomes more significant as the loading content of nano-oxide increases. For instance, it was observed that in the loading contents of 1% and 1.5wt% nano-zinc oxide, degradation rate was actually lower than that of the sample with no nano-zinc oxide. However, little oxidation occurred as confirmed by the presence of absorption peak at 1730cm^{-1}, associated with non-conjugated C=O groups, which is probably due to the formation of aldehyde and ketone groups. On the other hand, the degradation rate of wood as a consequence of the degradation of lignin aromatic rings was relatively low in the sample treated by pristine polyvinyl butyral solution as compared with the samples treated by zinc oxide loaded PVB solution, yet the oxidation can be detected in 1730cm^{-1}.

Conclusion

Synthetic resins have been employed as consolidate materials for the conservation of historical and cultural heritage. These resins may have some shortcomings which can be diminished using several methods particularly the use of mineral nano-materials. In this study, composites of PVB solution with 1% and 0.5 wt% nano-zinc oxides represented optimum results in protection of wooden samples especially from the viewpoints of water Absorption and wetting capability. By increasing the loading content of nano zinc oxide up to 1.5wt%, the hydrophilicity of wood was increased compared to the samples treated by pristine polyvinyl butyral (without nano zinc oxide). The accelerated aging test revealed that wood degradation was occurred in all samples except for A3. Thus, it can be deduced that loading low amounts of nano zinc oxide in polyvinyl butyral polymer matrix (0.5 – 1.0wt %) provided a low resistance under the accelerated aging condition. Also, it was observed that degradation rate in the samples treated with PVB solution containing 1.5 wt% zinc oxide was lower than other samples. Therefore, regarding the author's observation and findings, the loading amount of 1.0wt% nano-zinc oxide in polyvinyl butyral polymer matrix is the optimum content for preparation of an efficient consolidates for protection of dry historical woods against water.

Acknowledgments

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